# Reaction of Ketene with Acetone

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(1) Reaction temperature: Low—cooling with ice (0-10°C);
High—warming on a water bath (60-90°C).
(2) C: C-acetyl derivative of the compd. cont. A. M. H.
O: O-acetyl derivative of the compd. cont. A. M. H.
(3) ×: No reaction.
(4) Yield I: Yield based on the compd. cont. A. M. H. used.
II: Theoretical yield.
(5) Na-salt: Treated with 20 % $H_2SO_4$ after the reaction of ketene with the salt of the compd. cont. A. M. H.
(6) C(?): Not identified.

(4) With pyridine, a product rich in O-acetyl derivative and poor in C-acetyl derivative was obtained from acetylacetone or ethylacetoacetate, but it seemed the others gave solely C-acetyl derivatives.

The data of experiments and some physical constants of C-acetyl derivatives are summarized in Tables 1 and 2.

Table 2. Some physical constants of C-acetyl derivatives obtained from the compounds containg active methylenic hydrogen.

| C-acetyl derivative | Physical properties | | |
|---|---|---|---|
| | B. P. (°C) | $n_D$ | M. P. of Cu-salt (°C) |
| $CH_3COCH(COCH_3)_2$ | 100-102 (20mm) | $1.4872^{16}$ | decomp. (192-195) |
| $CH_3COCH(COCH_3)(COOC_2H_5)$ | 102-104 (20mm) | $1.4710^{15}$ | 150-1 |
| $CH_3COCH(COOC_2H_5)_2$ | 125 (14mm) | $1.4469^{13}$ | 122-3 |
| $CH_3COCH(CN)(COOC_2H_5)$ | 106-111 (20mm) | $1.4488^{23}$ | 234-5 |

## 9. Reaction of Ketene with Acetone

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In the reaction of ketene with acetone, β,β-dimethylacrylic acid $(CH_3)_2C{=}CHCOOH$ was obtained, when catalyzed with $BF_3$ and in the presence of conc. $H_2SO_4$, isopropenylacetate $(CH_3)(CH_2){>}COCOCH_3$ was produced. However, ketene did not react with sodium salt of acetone to give acetylacetone.

Some results of the experiments are as follows:

(1) Fifty grams of ketene was passed during 100 minutes into a solution of 1 g. of $BF_3$ in 58 g. of acetone at 0°C. Without removing $BF_3$, the product was distilled to give 32 g. of $\beta,\beta$-dimethylacrylic acid. Under similar conditions, using $(C_2H_5)_2O \cdot BF_3$ or $(CH_3CO)_2O \cdot BF_3$ as catalyst, 10 g. or 21 g. of $\beta,\beta$-dimethylacrylic acid was obtained.

With $AlCl_3$ of $ZnCl_2$, $\beta,\beta$-dimethylacrylic acid was not gained at all and the high polymers of ketene seemed to be produced. In the presence of $ZnCl_2$, a small amount of dehydroacetie acid was obtained.

(2) Forfy two grams of ketene was passed into a solution of 116 g. of acetone and 1 g. of conc. $H_2SO_4$ at about 50°. The product was fractionated to give 28 g. of isopropenylacetate. From a reaction of 59 g. of ketene, 87 g. of acetone and 1 g of conc. $H_2SO_4$, 30.5 g. of isopropenylacetate was obtained.

(3) On treating the reaction product with 20 % $H_2SO_4$ after the reaction of ketene with sodium salt of acetone was finished, acetylacetone was not obtained and pinaconelike substance was found, but it was not studied further.

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## 10. Mechanism to Improve the Crease Recovery of Fabrics by the Resin Treatment

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The degree of the crease recovery of fabrics, especially that of the spun rayon fabrics, is remarkably improved by the urea formaldehyde resin treatment, but in many cases, the degree of elasticity (elastic recovery) of the single fiber does not change so much as the crease recovery of the fabric. To make clear this relation, the degree of elasticity of the single fiber taken out from the resin treated spun rayon Serge and rayon Habutae was compared with the crease recovery of these fabrics. Moreover the degree of elasticity of the single fiber and zigzag-formed rayon yarn[1], and the crease recovery of the spun rayon Muslin, which have been treated by urea resin in various conditions, were estimated. From these results, it was found that the change of the degree of elasticity of the single fiber was small, but the degree of elasticity of zigzag-formed yarn and the crease recovery of the fabric remarkably increased almost in the same degree.

It is presumed that the recovery of deformation of the zigzag form of yarn constructing the fabric plays the main part to improve the crease resistance of fabrics by the resin treatment.

Following table gives some of the results obtained.